- (1) Iron; Fe; [7439-89-6]
- (2) Mercury; Hg; [7439-97-6]

EVALUATOR:

C. Guminski; Z. Galus Department of Chemistry University of Warsaw Warsaw, Poland

July, 1985

CRITICAL EVALUATION:

Early reports of the solubility of iron in mercury in the region of room temperature varied over a range from 4.1 x 10^{-17} to 6.39 at % (1-4). These results are all rejected because they are either much too low or too high as compared to recent more precise measurements. In some instances only the solubility limits were stated because the analytical methods could not detect the low solubility of iron near room temperature; the solubility limits reported varied from 10^{-6} to 10^{-3} at % in this temperature region (5-8). Palmaer (9) employed analytical methods and reported that the iron content in saturated iron amalgams remained nearly constant at about 2.5 x 10^{-4} at % between 293 and 484 K; this result is too high and is rejected. Kozin's (28) calculated solubility of 1.4×10^{-4} at % at 298 K is too high.

Marshall and coworkers (10) determined the solubility of iron between 298 and 973 K, and these authors observed an increase from 5.4×10^{-6} to 3.4×10^{-4} at %, respectively, in this temperature range. The data of (10) at temperatures below 700 K are clearly overstated, while the data at temperatures higher than 700 K are in good agreement with the subsequent works of Weeks and coworkers (11-14).

Weeks (11) graphically summarized the iron solubility determinations made at the Brookhaven National Laboratories by he and his coworkers (12-14). Numerical data were reported only at 873 and 923 K where the solubilities were 1.8 x 10^{-4} (12) and 2.7 x 10^{-4} at % (14), respectively. Earlier, preliminary results by these workers (15,16) are rejected because of the large scatter in the data. Nerad (17), without giving any experimental details, reported iron solubilities of 6.1 x 10^{-5} and 1.5 x 10^{-4} at % at 755 and 856 K, respectively; these solubilities are in good agreement with (10) and (11).

Wang (18) reported a solubility of 2.0×10^{-4} at % at 644 K, and Bowersox and Leary (19) determined a value of 5×10^{-5} at % at 623 K. Both these results are higher than the solubilities reported by (10); the result of (18) is rejected because it is too high as compared to the other measurements.

Parkman (20), using iron from two different sources, determined the iron solubility at several temperatures and at different equilibration times, but no definite conclusions may be made from the results of this study. Jangg and coworkers (21) reported that the iron content in saturated amalgams between 973 and 1073 K was less than 2×10^{-4} at %.

Because the scatter in the iron solubility data is large, it is difficult to make clear recommendations for the solubilities of this metal in mercury. There is an especial need for more precise measurements at temperatures below 573 K. Luborsky (22) found that a gel-like iron amalgam, which contained 1% Fe, was stable for long periods at room temperature, even though the apparent solubility was exceeded by more than a millionfold. In this instance, the particle size of the iron is about 2 nm in diameter and filtration through sintered glass does not appreciably change the composition. This formation of very fine crystallites of iron in the amalgam is the reason why almost all solubility determinations at the lower temperatures are strongly overstated.

Horsley (23) analyzed the data of (10) and reported iron solubility of (0.27-6.8) x 10^{-4} at % between 673 and 1073 K. This author also calculated grain boundary solubilities of (1.5-13.6) x 10^{-4} at % in this temperature range.

Gudtsov and Gavze (24, 25) investigated the solubility of steels in mercury, and they reported the content of iron in the mercury phase after hundreds of hours of contact at 673 to 1023 K. The authors found no evident dependence of the solubility on temperature, time of contact, or the composition of the steel; they reported solubilities ranging from $(0.109-8.4) \times 10^{-4}$ at %. Similar experiments were performed by Smith and Thompson (26) and by Parkman (20, 27). The solubilities obtained by (24, 25) are significantly higher than the solubility of pure iron; e.g., 6.2 x 10^{-2} at % for technical iron at 923 K as compared to 1.7 x 10^{-4} at % (11). On the other hand, (20, 27) found the solubility of technical iron to be of similar magnitude as that for pure iron.

Iron does not form any intermetallic compounds with mercury, and pure iron is in equilibrium with the liquid phase (8, 21, 24, 29).

(Continued next page)

- (1) Iron; Fe; [7439-89-6]
- (2) Mercury; Hg; [7439-97-6]

EVALUATOR:

C. Guminski; Z. Galus Department of Chemistry University of Warsaw Warsaw, Poland

July, 1985

CRITICAL EVALUATION: (Continued)

Tentative values of iron solubility in mercury:

T/K	Soly/at %	Reference
673	4×10^{-5}	[10]
773	$9 \times 10^{-5^{a}}$	[10,11,12]
873	2×10^{-4}	[11]
973	3.5×10^{-4}	[10]

a Interpolated value from data of cited references.

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- (1) Iron; Fe; [7439-89-6]
- (2) Mercury; Hg; [7439-97-6]

ORIGINAL MEASUREMENTS:

Marshall, A.L.; Epstein, L.F.; Norton, F.J. J. Am. Chem. Soc. 1950, 72, 3514-16.

VARIABLES:

PREPARED BY:

Temperature: 25-700°C

C. Guminski; Z. Galus

EXPERIMENTAL VALUES:

		Experim	ental S	Solubili	ty of	Iron in	Mercury				
t/°C						425					
g Fe/10 ⁶ g Hg	0.013	0.015	0.017	0.037	0.066	0.105	0.105	0.225	0.270	1.0	1.2

Smoothed Solubility of Iron in Mercury

t/°C			200					
g Fe/10 ⁶ Hg	0.015	0.019	0.030	0.054	0.11	0.21	0.45	0.96
^a Soly/at % x 10 ⁵	0.54	0.68	1.1	1.9	3.9	7.5	16	34

^aby compilers.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Carefully deoxygenated iron cylinder was equilibrated with Hg in evacuated quartz bulbs for several hours to a month. Care was taken to assure wetting of iron. Bulb was sealed in a steel bomb with Hg to equalize pressure at high temperatures, and the bomb was rocked in the furnace to assure equilibrium. Finally, the bomb was tilted at temperature to separate the amalgam from the iron, and then cooled to remove the sample for analysis. After opening the bulb the iron was removed and the Hg distilled, collected and weighed. The iron was determined colorimetrically by complex formation with KCNS.

SOURCE AND PURITY OF MATERIALS:

Redistilled Hg of original high purity, and pure Swedish iron were used.

ESTIMATED ERROR:

Soly: precision as high as \pm 50%.

Temp: nothing specified.

304 Iron

COMPONENTS: (1) Iron; Fe; [7439-89-6] (2) Mercury; Hg; [7439-97-6] VARIABLES: Temperature: 500-650°C ORIGINAL MEASUREMENTS: 1. Weeks, J.R. Corrosion 1967, 23, 98-106. 2. Weeks, J.R.; Minardi, A.; Fink, S. U.S. At. Ener. Comm. Rep., BNL-759, 1962, p. 63. PREPARED BY: C. Guminski; Z. Galus

EXPERIMENTAL VALUES:

The mass % solubility was presented graphically as a function of temperature. The data points from the plot were read off and converted to atomic % by the compilers.

<u>t/°C</u>	Soly/mass % x 10 ⁵	Soly/at $\% \times 10^4$
500	2.0	0.72
525	7.0	2.5
550	2.2	0.79
5 7 5	2.6	0.93
600	5.5	2.0
625	6.2	2.2
650	4.8	1.7

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Hg and Fe were placed into the larger chamber of a fused quartz capsule which was constructed so that a coarse quartz filter separated the two chambers. The capsule containing the metals was sealed under vacuum and placed in a stainless-steel capsule. Hg was also placed in the steel capsule before it was welded shut by tungsten-inert-gas welding. The capsule was placed in a furnace of a high temperature centrifuge and the sample was equilibrated for 72 hours. The sample was centrifuged after this period and the sat. amalgam was collected in the smaller quartz chamber. Hg from the weighed amount of amalgam was distilled off and the residue dissolved in HNO3-HF or aqua regia. Co or Y was added to the solution as internal standard and the Fe was determined spectrographically.

SOURCE AND PURITY OF MATERIALS: Mercury was triple-distilled, r

Mercury was triple-distilled, reagent grade.

Iron source and purity not specified, but specimens were first irradiated in the Brookhaven Graphite Research Reactor.

ESTIMATED ERROR:

Soly: nothing specified.

Temp: precision \pm 2 K.

REFERENCES ·

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Iron; Fe; [7439-89-6] (2) Mercury; Hg; [7439-97-6]	Bowersox, D.F.; Leary, J.A. U.S. At. Ener. Comm. Rep., LAMS-2518, 1961.
VARIABLES:	PREPARED BY:
One temperature: 350°C	C. Guminski; Z. Galus

EXPERIMENTAL VALUES:

The solubility of iron in mercury at 350°C was reported to be 2 x 10^{-3} g Fe/dm 3 Hg. The corresponding atomic % solubility calculated by the compilers is 5 x 10^{-5} at %.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solubility was determined by immersing a weighed coupon of Fe into a known amount of boiling Hg and periodically measuring the coupon weight. The solubility of Fe was determined from the weight loss of the coupon.

SOURCE AND PURITY OF MATERIALS:

Mercury was triple-distilled material.

Iron purity not specified.

ESTIMATED ERROR:

Soly: detection limit of method was 1×10^{-3} g; precision may be no better than \pm 50%.

Temp: nothing specified.

306 Iron

COMPONENTS:

- (1) Iron; Fe; [7439-89-6]
- (2) Mercury; Hg; [7439-97-6]

ORIGINAL MEASUREMENTS:

Parkman, M.F.

Extended Abst., Electrothermics and Metallurgy Div., Vol. 2, No. 2, The Electrochemical Soc., New York, NY 1964, pp. 16-21.

VARIABLES:

Temperature: 855-896 K

PREPARED BY:

C. Guminski; Z. Galus

EXPERIMENTAL VALUES:

The mass % solubility data were presented graphically; the solubilities were read off the curve and converted to atomic % by the compilers.

T/K	Fe Source	Contact time, hrs.	Soly/mass %	Soly/at %
855	Armco	16	9×10^{-6}	3.6×10^{-5}
855	Armco	16	1.5×10^{-5}	6.0×10^{-5}
866	Westinghouse	64	2.8×10^{-5}	1.1×10^{-4}
896	Westinghouse	132	2.2×10^{-5}	8.8×10^{-5}

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Specimen of Fe was placed in contact with Hg in a glass capsule and the Hg in the capsule was outgassed for at least 16 hours. The capsule was then sealed under vacuum. The capsule was placed in a copper block in a pressurized furnace and heated to the desired temperature and held there for 16 to 132 hours. A sample of the solution was then collected at temperature and cooled, and the Hg was separated from the sample by molecular distillation. The residue was dissolved into an acid solution and the Fe was determined by spectrophotometry.

SOURCE AND PURITY OF MATERIALS:

Iron from Armco contained 0.01% C, 0.03% Mn, 0.02% Si, 0.007% P, 0.04% S, 0.0012% O.

Iron from Westinghouse designated as
"Puron".

Mercury was probably triple-distilled.

ESTIMATED ERROR:

Soly: nothing specified.

Temp: precision + 3 K.

- (1) Ruthenium; Ru; [7440-18-8]
- (2) Mercury; Hg; [7439-97-6]

EVALUATOR:

C. Guminski; Z. Galus Department of Chemistry University of Warsaw Warsaw, Poland July, 1985

CRITICAL EVALUATION:

The few reports on the experimental determinations of the solubility of ruthenium in mercury are at wide variance. Strachan and Harris (1) reported a solubility of 0.694 at % at room temperature, but this result is clearly much too high. Jangg and Dörtbudak (2) could not detect any dissolution at 773 K; the detection limit for ruthenium by the latter authors was 2 x 10^{-5} at %. Bowersox and Leary (3) equilibrated the two metals at 293 and at 523 K, and they could not detect any dissolution of ruthenium at these temperatures. These authors concluded that the solubility was below their detection limit of 3 x 10^{-5} at %. It also was reported (4) that there was no attack of ruthenium by mercury at 823 K.

Dean (5) reported that the solubility of ruthenium is of the order of 2×10^{-7} at %, but the temperature and other experimental details were not specified. Messing and Dean (6) reported that the solubility of ruthenium in a saturated uranium amalgam varied from 2.4 x 10^{-3} at % at 323 K to 2.2 x 10^{-2} at % at 629 K.

Kozin predicted a solubility of 1.2 x 10^{-11} at % at 298 K (7); he previously predicted 9.3 x 10^{-17} at % at 298 K (8). The first value appears to be more reliable to the evaluators.

It is clear that there is no dependable solubility data in this system; it only may be stated that the solubility of ruthenium at 298 K is less than 2 x 10^{-5} at %.

The saturated amalgam is in equilibrium with pure ruthenium (2).

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COMPONENTS: (1) Ruthenium; Ru; [7440-18-8] (2) Mercury; Hg; [7439-97-6] VARIABLES: Temperature: 20-250°C ORIGINAL MEASUREMENTS: Bowersox, D.F.; Leary, J.A. U.S. At. Ener. Comm. Rep., LAMS-2518, 1961. PREPARED BY: C. Guminski; Z. Galus

EXPERIMENTAL VALUES:

The solubility of ruthenium in mercury at 20 and 250°C was reported to be less than the detection limit of 2 x 10^{-3} g of Ru in 1 dm³ of Hg. The corresponding atomic % detection limit calculated by the compilers is 3 x 10^{-5} at %. Although Ru apparently dissolved in Hg at 350°C, it did not pass through a coarse Pyrex frit at either 30 or 350°C. Therefore, since the "solubility is considered to be the quantity that passes through such a frit, Ru would, by definition, be insoluble in Hg."

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solubility was determined by immersing a weighed coupon of Ru into a definite amount of Hg at specified temperatures. The coupon weight was measured periodically to determine the solubility from the weight loss.

SOURCE AND PURITY OF MATERIALS:

Triple-distilled Hg was used.

Ruthenium purity was not specified.

ESTIMATED ERROR:

Soly: detection limit as specified above. Temp: not specified.

- (1) Osmium; Os; [7440-04-2]
- (2) Mercury; Hg; [7439-97-6]

EVALUATOR:

C. Guminski; Z. Galus Department of Chemistry University of Warsaw Warsaw, Poland July, 1985

CRITICAL EVALUATION:

Jangg and Dörtbudak (1), in an equilibration study at 773 K, could not detect any dissolution of osmium in mercury at their analytical detection limit of 10^{-5} at %. The low solubility of osmium is also suggested by the estimate reported by Kozin (2) of 1.1 x 10^{-14} at % at 298 K. The latter value appears to the evaluators to be more reliable than one predicted previously, i.e., 1.8 x 10^{-22} at % at 298 K (3).

The saturated osmium amalgam is in equilibrium with pure osmium; no Os-Hg compounds were found (1).

References

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 Kozin, L.F. Fiziko-Khimicheskie Osnovy Amalgamnoi Metallurgii, Nauka, Alma-Ata, <u> 1964</u>.
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